

JAN 22 1979

pten 830-H-15 NASA 60:1379

NASA Technical Paper 1379

COMPLETED
ORIGINAL

Lubrication and Failure Mechanisms of Molybdenum Disulfide Films

II - Effect of Substrate Roughness

Robert L. Fusaro

DECEMBER 1978

NASA

30

NASA Technical Paper 1379

Lubrication and Failure Mechanisms of Molybdenum Disulfide Films

II - Effect of Substrate Roughness

Robert L. Fusaro
Lewis Research Center
Cleveland, Ohio



National Aeronautics
and Space Administration

**Scientific and Technical
Information Office**

1978

SUMMARY

The friction, wear, and wear life of rubbed molybdenum disulfide (MoS_2) films were studied in a pin-on-disk sliding friction and wear apparatus. The films were applied to 440C HT (high temperature modified) stainless-steel disks with three surface finishes - polished ($0.09 \pm 0.02 \mu\text{m CLA}$), sanded ($0.30 \pm 0.05 \mu\text{m CLA}$), and sandblasted ($1.2 \pm 0.2 \mu\text{m CLA}$). The films were evaluated in moist air (10 000-ppm H_2O) and dry argon (<20 -ppm H_2O). Optical microscopy was used to study the lubricating films, the transfer films, and the wear process. Observations were made at preset intervals throughout the wear lives of the films by stopping the tests and removing the specimens from the apparatus.

During "run-in," very smooth, flat plateaus were created on the rider and also on metallic asperities (on the rough substrate surfaces) in the MoS_2 film wear track. The lubrication mechanism was the flow of thin films of MoS_2 between the flat plateaus on the two opposing surfaces. The valleys on the roughened substrate surfaces served three purposes: They restricted the transverse flow of the MoS_2 out of the contact area during run-in, they acted as reservoirs for supplying MoS_2 to the contact area, and they acted as deposit sites for wear debris. Thus, in general, wear life was extended by increasing the surface roughness of the substrate; however, the friction coefficient was not markedly affected.

Failure was related to the depletion of MoS_2 from the contact zone. This depletion occurred in two ways. In moist air, metallic-colored, coalesced films of MoS_2 were transformed to a black, powdery material. X-ray diffraction studies showed that this material was mainly α -iron, MoO_3 , the possibly FeMoO_3 . In dry argon, there was no optical evidence of MoS_2 transformation, and failure was due mainly to the gradual depletion of the film by transverse flow from the contact area. X-ray diffraction analysis showed the powdery material on the wear track at failure in dry argon to be α -iron, MoS_2 , and possibly FeS .

The chemical transformation took place very rapidly in moist air; thus, wear life was about two orders of magnitude greater in dry argon than in moist air. Increasing the substrate roughness also extended wear life (in both atmospheres), but the effect was not as great as the atmosphere effect (less than one order of magnitude). The friction coefficient was not affected by substrate roughness in dry argon; however, in moist air, rougher substrates gave lower friction coefficients than smoother substrates. Rider wear rates during run-in were higher on rougher substrates for both atmospheres. After run-in in dry argon, substrate roughness did not greatly affect rider wear rate; however, after run-in in moist air, the rougher the substrate, the lower the rider wear rates.

INTRODUCTION

Molybdenum disulfide (MoS_2) is one of the most widely used solid lubricants. A considerable amount of research has been conducted on its friction properties and the reasons for its good lubricating behavior. Johnson (ref. 1), Winer (ref. 2), and Farr (ref. 3) have written excellent reviews on the history, the uses, and the fundamental knowledge of MoS_2 as a lubricant.

Many studies have been conducted on the lubrication mechanisms of MoS_2 films. Most of these studies have been on a molecular level; that is, they have related the good lubricating properties of MoS_2 to its hexagonal crystal structure and covalent nature (refs. 2 and 3) or to its highly polarized surface that adheres well to metals (ref. 4). Many studies have also been conducted on the failure mechanisms of MoS_2 films. Failure has been related to the chemical degradation of MoS_2 , with the most predominant chemical reaction in air being oxidation (refs. 5 to 12). It has also been proposed that water vapor accelerates the oxidation process (ref. 10).

Very few studies have been conducted on how MoS_2 films lubricate and fail from a microscopic point of view. In one of the few, Salomon, De Gee, and Zaat (refs. 8 and 9) have observed and recorded morphological changes of MoS_2 films photographically by stroboscopic illumination. They have observed that MoS_2 compacts and sinters on the wear track to form a highly reflective film and that oxidation of the MoS_2 leads to embrittlement, blistering, and scaling of the film.

In the first part of this study (ref. 13), the effects of oxygen and water vapor on lubrication, transfer, and failure were investigated. In general, it was found that the lubrication mechanism was the formation of thin, metallic-colored, coalesced MoS_2 films on each sliding surface and the plastic flow of these films between the sliding surfaces in relative motion. In air, the failure mechanism was found to be the transformation of the metallic-colored, coalesced MoS_2 films to a black, powdery material. Water in the air atmosphere appeared to accelerate this transformation rate. In argon, no transformation of MoS_2 was observed (with a microscope), but cracking and spalling of the coalesced MoS_2 film resulted in its gradual depletion.

The second part of this study (reported herein) investigated the effect of substrate surface roughness (1) on the lubrication mechanism; (2) on the failure mechanism and the chemical composition of the black, powdery material on the wear track; (3) on the wear life; (4) on the friction coefficient; and (5) on the rider wear rate. Rubbed MoS_2 films were applied to disk substrates that were polished ($0.09 \pm 0.02 \mu\text{m}$ CLA (centerline average)), sanded ($0.30 \pm 0.05 \mu\text{m}$ CLA), and sandblasted ($1.2 \pm 0.2 \mu\text{m}$ CLA). To separate any effects caused by oxidation of the MoS_2 film, experiments were conducted in both moist air (10 000-ppm H_2O) and dry argon (<20 -ppm H_2O).

The tests were stopped at preset sliding intervals and then the sliding surfaces were examined by optical microscopy at magnifications to 1100. A pin-on-disk sliding friction

apparatus and 440C HT steel specimens were used. The experimental conditions were a temperature of 25° C, a sliding speed of 2.6 meters per second (1000 rpm), and a load of 1 kilogram.

MATERIALS

Technical-grade MoS₂ powder with an average particle size of 10 micrometers was used in this study. The size ranged from less than 1 micrometer to 75 micrometers; however, the large particles appeared to be conglomerations of small particles.

The riders and disks were made of 440C HT stainless steel with a Rockwell hardness of C-58 to C-60. The disks were lapped and polished to a surface finish of 0.09±0.02 micrometer CLA (centerline average). Some disks were then roughened either by sanding them in random directions with number-150-grit wet sandpaper to 0.30±0.05 micrometer (CLA) or by sandblasting them to 1.2±0.2 micrometers (CLA).

The MoS₂ powder was applied to the polished or roughened disk surfaces by mechanically rubbing it over the surface at constant load (see section PROCEDURE). The thickness of the films obtained was estimated optically and by surface profilometry to be about 1 to 2 micrometers above the highest feature on the metallic surface.

APPARATUS

A pin-on-disk sliding friction apparatus was used in this study. This apparatus is described in reference 14. Basically the friction specimens (fig. 1) were a flat disk (6.3-cm diam) in sliding contact with a stationary hemispherically tipped rider (0.476-cm radius). The rider slid on a 5-centimeter-diameter track on the disk for a linear sliding speed of 2.6 meters per second at a disk rotation of 1000 rpm.

The apparatus used to apply the MoS₂ powder to the disks is shown in figure 2. The disk was attached to the vertical shaft of a small electric motor by means of a cup-shaped holder. Two vertical rods were used to restrain a floating metal plate to which were attached the MoS₂ applicators. In these experiments, the backs of polishing cloths were used as applicators. The rubbing load was applied by placing two 1-kilogram weights on top of the metal plate.

The application apparatus was designed to fit under the bell jar of a vacuum system. The atmosphere in which the films were applied could thus be controlled by evacuating the bell jar and backfilling it with the desired atmosphere. Previous results (ref. 15) have shown that films applied in moist air are thicker and more dense than films applied under dryer conditions. Thus the films used in these experiments were applied in moist air (10 000-ppm H₂O).

PROCEDURE

Surface Cleaning

The cleaning procedure was as follows:

- (1) Scrub surfaces under running tap water with a brush to remove abrasive particles.
- (2) Wash surfaces with pure ethyl alcohol.
- (3) Rub surface with a water paste of levigated alumina. Clean until water readily wets surface.
- (4) Rinse under running tap water to remove levigated alumina. (Use brush to facilitate removal.)
- (5) Rinse in distilled water.
- (6) Dry surface with dry compressed air, since surfaces not dried quickly tend to oxidize.

Film Application

The procedure for applying the rubbed films was as follows:

- (1) Apply a small amount of MoS_2 powder to the cleaned disk surface and spread it evenly over the surface with the back of a polishing cloth.
- (2) Apply approximately 1 gram of MoS_2 powder to the contact zone of the applicator (back of a polishing cloth attached to the floating metal plate) and distribute it evenly.
- (3) Assemble apparatus as shown in figure 2 and apply two 1-kilogram weights as the applied load.
- (4) Evacuate the bell jar to $1 \times 10^{-3} \text{ N/m}^2$ and backfill it with moist air (10 000-ppm H_2O) to atmospheric pressure. Continue to purge the bell jar with moist air until application is complete.
- (5) Set disk into rotation, gradually increase the speed to 15 rpm, and rub for 1 hour.
- (6) Remove disk from apparatus and blow off loose MoS_2 debris from the surface with dry compressed air.

Friction and Wear Tests

A rider and disk (with applied MoS_2 film) were inserted into the friction and wear apparatus, and the test chamber was sealed. Moist air (10 000-ppm H_2O) or dry argon (<20-ppm H_2O) were purged through the chamber for 15 minutes before the tests; this purge was continued throughout the tests. Moist air was used as a control atmosphere

since it is typical of average atmospheric conditions (approximately 50 percent relative humidity). Dry argon was used since it is inert and nonoxidizing.

The flow rate was 1500 cubic centimeters per minute, and the volume of the chamber was 2000 cubic centimeters. After the 15-minute purge, the disk was set into rotation at 1000 rpm and a 1-kilogram load was gradually applied. The test temperature was 25° C.

Each test was stopped after 1 kilocycle (1 min) of sliding. After the rider and disk were removed from the friction apparatus, the contact areas were examined by optical microscopy and photographed. Surface profiles of the film wear tracks were also taken. The rider and disk were then placed back into the apparatus, and the test procedure was repeated. The rider was not removed from the holder, and locating-pins in the apparatus insured that it was returned to its original position.

Each test was stopped and the test procedure repeated after sliding intervals of 1, 5, 15, 30, 60, 100, 200, 400, 700, 1500, 2700, and 3700 kilocycles or when failure occurred. The failure criterion in this study was a friction coefficient of 0.30; thus wear life is defined as the number of kilocycles of sliding to reach a friction coefficient of 0.30. Rider wear was determined by measuring the diameter of the wear scar on the hemispherically tipped rider and then calculating the volume of material worn away.

Analysis of Sliding Surfaces

Optical microscopy techniques were used to study the lubricating films, the transfer films, and the wear particles. The surfaces were viewed at magnifications to 1100. At these high magnifications, the vertical resolution was low ($\sim 1 \mu\text{m}$); this aspect was used to measure the heights of various features on the sliding surfaces, such as film thickness and wear track depth.

With vertical illumination of the surfaces, interference fringes could be seen in the films, both on the disk wear track and on the rider wear scar. Interference fringes indicated that the MoS_2 particles had flowed together to form a continuous film that was very smooth and that the film was being sheared thinner and thinner in some areas. For example, the gradual depletion of the fringes in the inlet area of the rider wear scar indicated that the film thickness was less than the wavelength of light ($0.4 \mu\text{m}$).

The powdery debris that remained on the disk wear tracks at failure was scraped off and mounted on a quartz fiber with apiezon. An X-ray diffraction pattern was then taken, by using the Debye-Scherrer method, to determine the composition.

RESULTS AND DISCUSSION

Moist-Air Results

The MoS_2 powder was rubbed onto 440C HT steel disks with three surface finishes: lapped and polished to a surface roughness of 0.09 ± 0.02 micrometer (CLA), sanded to a surface roughness of 0.30 ± 0.05 micrometer (CLA), and sandblasted to a surface roughness of 1.2 ± 0.2 micrometers (CLA). Figure 3 gives photomicrographs and surface profiles of the three substrate surfaces before the MoS_2 films were applied. Figure 4 shows photomicrographs and surface profiles after the MoS_2 films were applied to these surfaces.

The effect of substrate surface finish on friction coefficient and wear life in a moist-air test atmosphere ($10\,000\text{-ppm H}_2\text{O}$) is shown in figure 5. The gaps in the traces represent the intervals when the tests were stopped so that wear measurements could be made and the sliding surfaces could be observed with an optical microscope. The sandblasted surface gave a lower friction coefficient for a longer time than did either the sanded surface or the polished surface.

The failure criterion for solid-lubricant films is usually arbitrary. Most often, failure is assumed as the point when the friction coefficient reaches some predetermined value or rapidly increases in value. In these experiments, failure was deemed to occur when the friction coefficient reached 0.30. This occurred for the polished surface after $3\frac{1}{2}$ kilocycles of sliding, for the sanded surface after $5\frac{1}{2}$ kilocycles, and for the sandblasted surface after 60 kilocycles. Sliding was continued past this point to determine how friction and rider wear were affected. The friction coefficient increased beyond 0.30 and then dropped below 0.30 (fig. 5).

At the preset sliding intervals, the tests were stopped, the rider and disk specimens were removed from the apparatus, the rubbing surfaces were examined, and the wear to the riders was calculated. Table I gives rider wear rates for each sliding interval. The rougher the substrate to which the MoS_2 was applied, the greater the initial wear to the rider. After 1 kilocycle of sliding on MoS_2 films applied to polished, sanded, and sandblasted substrates the rider wear rates were 1.8×10^{-15} , 2.0×10^{-15} , and 8.8×10^{-15} , respectively.

Figure 6, which gives photomicrographs of rider wear scars and MoS_2 film wear tracks after 1 kilocycle of sliding, illustrates the difference in rider wear and transfer. Figure 7 gives high-magnification photomicrographs of the central area of the MoS_2 film wear tracks; figure 8 gives high-magnification photomicrographs of the transfer films on the riders.

As shown in figures 6 and 7, MoS_2 did not produce a continuous film on the disk wear track, regardless of the substrate surface finish to which the MoS_2 was applied. On the polished substrate surface, individual platelets of MoS_2 formed and tended to

coalesce, but the density was not sufficient to cover the surface (fig. 7(a)). Excess MoS_2 either flowed out of the wear track at the sides or coalesced to form clumps (fig. 6(a)). The MoS_2 tended to fill up the scratches on the wear track of the sanded substrate surface (fig. 7(b)), and the area between the scratches was either void of MoS_2 or covered with a film too thin to observe. The amount of MoS_2 on the wear track of the sandblasted substrate surface appeared to be greater than for the other two surfaces; however, the film still did not cover the wear track completely. "Flats" on the metallic asperities can be seen in the wear track (fig. 7(c)). These flat plateaus were formed during the initial run-in phase of sliding by the interaction between the sharp sandblasted asperities on the substrate and the rider. This interaction was the most likely reason for the greater initial rider wear on the sandblasted substrate surface. Once flats had formed on the sandblasted asperities (sliding time greater than 1 kilocycle), rider wear was less for the sandblasted substrate surface than for the other two surfaces (table I). Friction was lower because the valleys in the sandblasted substrate surface served as reservoirs for the MoS_2 and restricted its transverse flow from the contact zone.

Heavy, coalesced transfer films of MoS_2 were found after 1 kilocycle of sliding on the riders that slid on the films applied to the polished and sanded substrate surfaces. However, the transfer was not nearly as thick or continuous to riders that slid on films applied to the sandblasted substrate surface (figs. 6 and 8) probably because of the scouring action of the sharp metallic asperities on this surface. This thin transfer of MoS_2 did not increase the friction coefficient; in fact, a slightly lower friction coefficient was obtained.

With increasing sliding time, a feature common to all wear tracks and rider transfer films was a distinct change in the appearance of the MoS_2 film. Initially, the MoS_2 on the wear track was a bright, metallic-colored, coalesced film. As sliding progressed, the MoS_2 films on the wear tracks turned black and were no longer smooth and coalesced. The rate of this transformation seemed to be related to the roughness of the substrate surface.

Figure 9 shows high-magnification photomicrographs of the wear tracks on MoS_2 films applied to a sandblasted substrate surface after 5, 15, 60, and 70 kilocycles of sliding. As sliding time increased, more MoS_2 was transformed. Because more wear also occurred on the flat plateaus on the sandblasted asperities, new lubricant was exposed deeper in the valleys. Thus, the valleys in the rough surface served as reservoirs for the lubricant and as deposit sites for wear particles.

At failure, the film wear tracks and the rider wear scars looked much the same (fig. 10). There was a heavy, powdery buildup of transformed material on both these surfaces. The transformed material did not completely cover the wear surfaces but formed bands around the circumference of the disk wear track and parallel to the sliding direction on the rider wear scar. The powdery material on the wear track of the sanded disk was analyzed by X-ray diffraction and no MoS_2 was found. The powdery material

consisted of α -iron, MoO_3 , and possibly FeMoO_3 . This agrees with the optical observations of MoS_2 transforming to another chemical species.

Each test was continued past the arbitrarily chosen failure point - a friction coefficient of 0.30. In each case both the friction coefficient (fig. 5) and the rider wear rate (table I) decreased. Thus either the transformed MoS_2 was providing lubrication, or MoS_2 at the sides of the wear track or deeper in the valleys (of the rough surfaces) had found its way into the contact region. The small reductions in friction coefficient and rider wear rate for the MoS_2 film applied to the polished surface suggest that the main contribution probably came from exposure of new MoS_2 (from deeper in the valleys of the rough surfaces).

Dry-Argon Results

In the first part of this investigation (ref. 13), it was shown that the transformation of MoS_2 on the sliding surfaces did not occur in dry argon (<20 -ppm H_2O). Thus, a series of experiments was also conducted in dry argon to determine the effect of substrate surface roughness on the lubricating properties of MoS_2 films when transformation of MoS_2 was not the principal mechanism of failure. Friction traces for these tests (fig. 11) illustrate that the friction coefficient was not affected by the roughness of the substrate - the value being slightly less than 0.02 for the first 700 cycles. Wear life, however, was affected, just as it was in moist air; that is, the rougher the surface, the longer the wear life. Even on the polished substrate, wear life was much greater in dry argon than in moist air. The wear lives obtained were 950 kilocycles for the polished substrate, 1860 kilocycles for the sanded substrate, and 4450 kilocycles for the sandblasted substrate.

Table II gives rider wear rates in dry argon for each sliding interval. As in moist air, the rougher the substrate, the greater the initial rider wear. However, the wear rate was lower in dry argon. The values obtained after 1 kilocycle of sliding were $0.10 \times 10^{-15} \text{ m}^3/\text{m}$ for the polished substrate, $0.30 \times 10^{-15} \text{ m}^3/\text{m}$ for the sanded substrate, and $4.5 \times 10^{-15} \text{ m}^3/\text{m}$ for the sandblasted substrate. After run-in the rates dropped considerably, with the polished surface giving the lowest rate ($0.002 \times 10^{-15} \text{ m}^3/\text{m}$). The sandblasted substrate gave a low rate of $0.003 \times 10^{-15} \text{ m}^3/\text{m}$, and the sanded substrate gave a low rate of $0.008 \times 10^{-15} \text{ m}^3/\text{m}$.

The wear and transfer to the riders after 1 kilocycle of sliding in dry argon can be compared for the three surface finishes in figure 12. As in moist air, the smoother the substrate surface, the greater the transfer to the rider and the less the wear. Even though transfer and rider wear were different at this point, the friction coefficients were the same. After 15 kilocycles of sliding, the transfer films for riders on the polished

and sanded substrates became like those for riders on the sandblasted substrate after 1 kilocycle of sliding (fig. 12(c)). That is, very thin films of MoS_2 were found on the rider scar.

Figure 13 gives high-magnification photomicrographs of the MoS_2 film wear tracks after 1 kilocycle of sliding in dry argon. On the polished and sandblasted substrates the MoS_2 film was not continuous; however, on the sanded substrate a more continuous film was produced. Because the longest lives were obtained on the sandblasted substrate, film continuity was not the most important factor in determining wear life.

In the first part of this investigation (ref. 13), it was found that for MoS_2 films applied to sanded substrates, the failure mechanism was the cracking and spalling of the continuous, metallic-colored MoS_2 films. This, coupled with the tendency of the MoS_2 to flow transversely outward on the film wear track, gradually depleted the films. For films applied to polished substrates, very little cracking or spalling was observed. The failure mechanism seemed to be solely the depletion of the coalesced MoS_2 films by transverse flow. Figure 14 gives high-magnification photomicrographs of the wear tracks on the MoS_2 films applied to a polished substrate after 60, 700, and 940 kilocycles of sliding. The thinning and gradual depletion of the films can be seen by comparing the surfaces at 60 and 700 kilocycles of sliding. When the films became too thin, metal-to-metal contact occurred and fine, powdery metallic debris was produced. If only a small amount of debris was produced, the films could reheal and only a spike in the friction coefficient trace resulted (fig. 11). However, at some point, the production of fine metallic debris became too great, causing the friction to increase and bands of powdery material to be created around the wear track. Figure 14(c) shows this powdery buildup on the wear track at failure.

On the sandblasted substrate, the MoS_2 film on the wear track tended to build up around the highest metallic asperities. Figure 13(c) shows a typical region on the track. Smooth, compacted MoS_2 can be seen along with flat, metallic asperity plateaus. As in moist air, the interaction of the sharp, sandblasted asperities (during run-in) with the rider was the most likely reason for the greater initial rider wear when sliding on the sandblasted substrate. After run-in the lubrication mechanism was the plastic flow of the MoS_2 films between the flat asperity plateaus and the flat rider scar. As sliding progressed, the MoS_2 in the contact region was gradually depleted, causing the metallic asperity plateaus to wear and thereby exposing new lubricant deeper in the valleys.

Figure 15 shows a high-magnification photomicrograph that illustrates the lubrication mechanism of MoS_2 films applied to sandblasted substrates. The photomicrograph, taken after 1700 kilocycles of sliding, shows a flat, metallic plateau with thin films of MoS_2 flowing over it. The MoS_2 is supplied from the valleys, becomes compressed in the entrance region of the plateau, and eventually coalesces into a very thin film as it flows across the plateau and then is deposited in a following valley. The same process occurs on the rider, which in effect is also a flat, metallic plateau - but much larger.

The MoS_2 builds up in the entrance region of the rider scar, compresses, and eventually coalesces as it flows across the rider scar and is deposited in the exit region (fig. 12).

At failure, the film wear tracks on the sandblasted substrate and the rider wear scars looked much the same as they did for the other two substrates. That is, there was a heavy buildup of powdery material on both surfaces. This powdery material was scraped off the disk wear track and analyzed by X-ray diffraction. No molybdenum oxides were found for any test conducted in dry argon, and the powdery material was mostly α -iron. Also found were a few weak MoS_2 lines and possibly some FeS lines, although the FeS lines were too weak for good analysis. Thus, in dry argon, failure was deemed to be caused by the gradual depletion of MoS_2 and the consequent excessive production of very fine metallic wear debris.

SUMMARY OF RESULTS

Friction, wear, and optical microscopy studies of molybdenum disulfide (MoS_2) rubbed films applied to 440C HT steel with different substrate surface finishes and then evaluated in moist air (10 000-ppm H_2O) or dry argon (<20-ppm H_2O) gave the following results:

1. The lubricating mechanism consisted of the plastic flow (shear) of thin films of MoS_2 between flat plateaus on the rider and on the metallic substrate. If the substrate was rough, flat plateaus were created during run-in. The MoS_2 tended to flow across these flat plateaus.
2. Failure in moist air was due to the transformation of metallic-colored, coalesced films of MoS_2 to a black, powdery material that was found by X-ray diffraction to be α -iron, MoO_3 , and possibly FeMoO_3 .
3. Failure in dry argon was caused by the gradual depletion of MoS_2 by lateral flow from the contact region and the consequent production of very fine powdery debris that was found by X-ray diffraction to be mostly α -iron. A very small amount of residual MoS_2 and possibly FeS was also found in the powder, but no molybdenum oxides were found.
4. Wear life was about two orders of magnitude greater in dry argon than in moist air because of the difference in failure mechanisms. Increasing the substrate surface roughness tended to extend wear life (both in moist air and in dry argon) by providing reservoirs for the MoS_2 .
5. The friction coefficient was not affected by substrate roughness in dry argon; however, in moist air, rougher substrates gave lower friction coefficients than smoother substrates.

6. Rougher substrates caused higher wear rates during run-in in both moist air and dry argon. After run-in, rider wear rates in dry argon were not greatly affected by substrate roughness; however, in moist air, the rougher the substrate, the lower the rider wear rates.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 14, 1978,
505-04.

REFERENCES

1. Johnson, R. L.: A Review of the Early Use of Molybdenum Disulfide as a Lubricant. Nat. Lubric. Grease Inst. Spokesman, vol. 32, no. 8, Nov. 1968, pp. 298-305.
2. Winer, W. O.: Molybdenum Disulfide as a Lubricant: A Review of the Fundamental Knowledge. Wear, vol. 10, 1967, pp. 422-452.
3. Farr, J. P. G.: Molybdenum Disulfide in Lubrication. A Review. Wear, vol. 35, 1975, pp. 1-22.
4. Holinski, R.; and Günsheimer, J.: A Study of the Lubricating Mechanism of Molybdenum Disulfide. Wear, vol. 19, 1972, pp. 329-342.
5. Peterson, Marshall B.; and Johnson, Robert L.: Friction and Wear Investigation of Molybdenum Disulfide. I - Effect of Moisture. NACA TN-3055, 1953.
6. Ross, Sydney; and Sussman, Alan: Surface Oxidation of Molybdenum Disulfide. J. Phys. Chem., vol. 59, Sept. 1955, pp. 889-892.
7. Haltner, A. J.; and Oliver, C. S.: Effect of Water Vapor on the Friction of Molybdenum Disulfide. Ind. Eng. Chem. Fundam., vol. 5, Aug. 1966, pp. 348-355.
8. Salomon, G.; De Gee, A. W. J.; and Zaat, J. H.: Mechano-Chemical Factors in MoS₂ Film Lubrication. Wear, vol. 7, Jan.-Feb. 1964, pp. 87-101.
9. De Gee, A. W. J.; Salomon, G.; and Zaat, J. H.: On the Mechanisms of MoS₂ Film Failure in Sliding Friction. Trans. ASLE, vol. 8, Apr. 1965, pp. 156-163.
10. Pritchard, C.; and Midgley, J. W.: The Effect of Humidity on the Friction and Life of Unbonded Molybdenum Disulfide Films. Wear, vol. 13, 1969, pp. 39-50.
11. Pardee, Robert P.: The Effect of Humidity on Low-Load Frictional Properties of a Bonded Solid Film Lubricant. Trans. ASLE, vol. 15, no. 2, 1972, pp. 130-142.

12. Gänshheimer, J.: A Review on Chemical Reactions of Solid Lubricants during Friction. Trans. ASLE, vol. 15, no. 4, 1972, pp. 244-251.
13. Fusaro, Robert L.: Lubrication and Failure Mechanisms of Molybdenum Disulfide Films. I - Effect of Atmosphere. NASA TP-1343, 1978.
14. Fusaro, Robert L.; and Sliney, Harold E.: Graphite Fluoride as a Solid Lubricant in a Polyimide Binder. NASA TN D-6714, 1972.
15. Johnston, R. R. M.; and Moore, A. J. W.: The Burnishing of Molybdenum Disulphide onto Metal Surfaces. Wear, vol. 7, Nov. -Dec. 1964, pp. 498-512.

TABLE I. - RIDER WEAR RATES IN A MOIST-AIR

ATMOSPHERE (10 000-ppm H₂O)

[Experimental conditions: rubbed MoS₂ films;
440C HT steel riders and disks; temperature,
25⁰ C; load, 1 kg; speed, 2.6 m/sec (1000 rpm).]

Sliding interval, kilocycles	Sandblasted substrate	Sanded substrate	Polished substrate
	Surface finish, μm		
	1.2 \pm 0.2	0.30 \pm 0.15	0.09 \pm 0.02
	Rider wear rate, m ³ /m		
0 - 1	8.80 $\times 10^{-15}$	2.00 $\times 10^{-15}$	1.8 $\times 10^{-15}$
1 - 5	.64	.55	^a 3.6
5 - 10	-----	^a 2.40	2.8
5 - 15	.07	-----	-----
10 - 30	-----	.49	-----
15 - 60	.11	-----	-----
60 - 70	^a 1.60	-----	-----
70 - 100	.22	-----	-----

^aFailure.

TABLE II. - RIDER WEAR RATES IN A DRY-ARGON

ATMOSPHERE (<20-ppm H₂O)

[Experimental conditions: rubbed MoS₂ films;
440C HT steel riders and disks; temperature,
25⁰ C; load, 1 kg; speed, 2.6 m/sec (1000 rpm).]

Sliding interval, kilocycles	Sandblasted substrate	Sanded substrate	Polished substrate
	Surface finish, μm		
	1.2 \pm 0.2	0.30 \pm 0.15	0.09 \pm 0.02
	Rider wear rate, m ³ /m		
0 - 1	4.500 $\times 10^{-15}$	6.490 $\times 10^{-15}$	0.100 $\times 10^{-15}$
1 - 15	.048	.062	.038
15 - 60	.013	.019	.018
60 - 200	.009	.012	.004
200 - 400	.003	.012	.002
400 - 700	.006	.008	.002
700 - 940	-----	-----	^a .010
700 - 1500	.010	.011	-----
1500 - 1860	-----	^a .018	-----
1500 - 2700	.012	-----	-----
2700 - 3700	.009	-----	-----
3700 - 4450	^a .046	-----	-----

^aFailure.

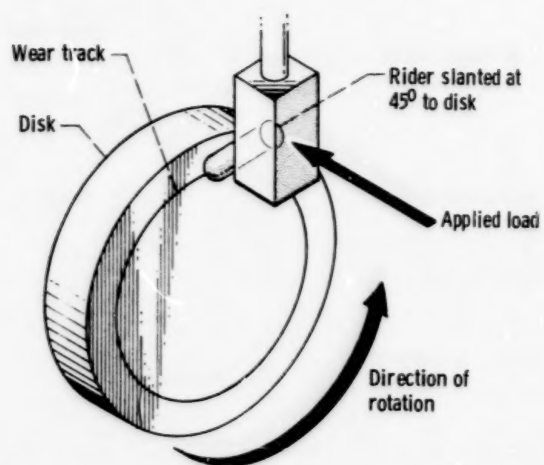


Figure 1. - Schematic diagram of friction specimens.

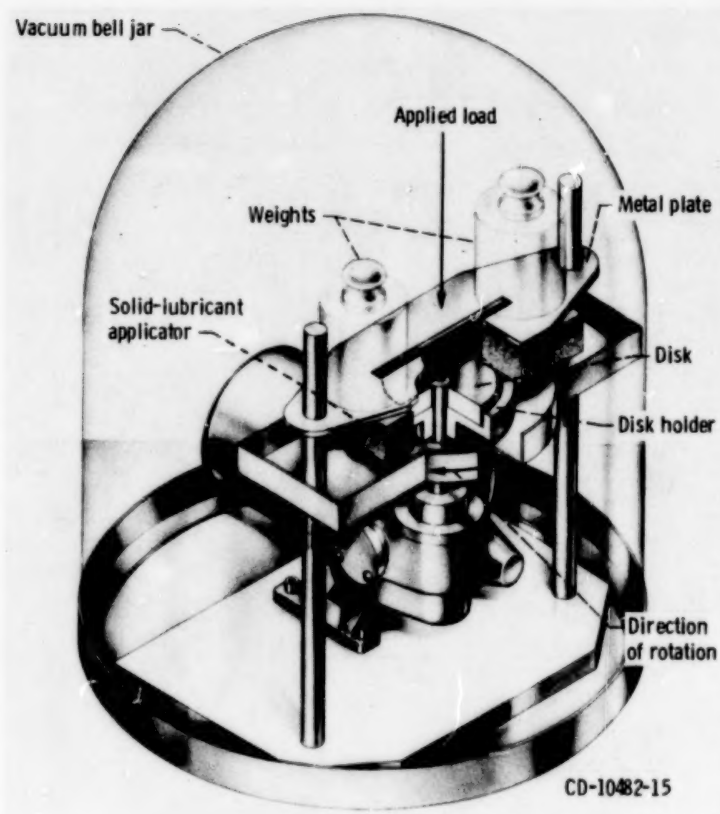
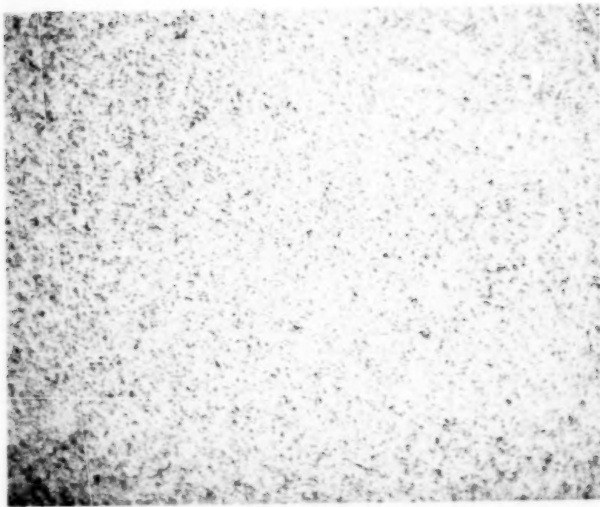
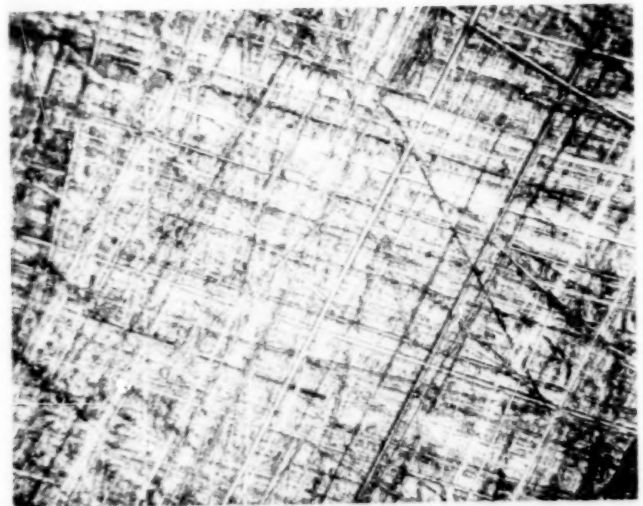


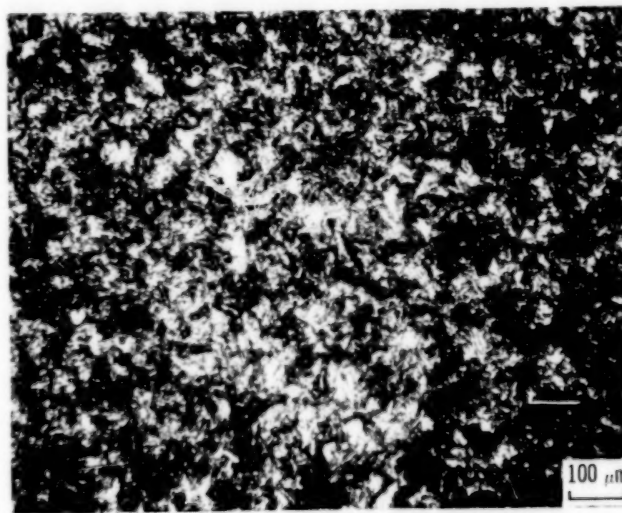
Figure 2. - Apparatus used to apply MoS_2 films.



(a) Polished substrate.

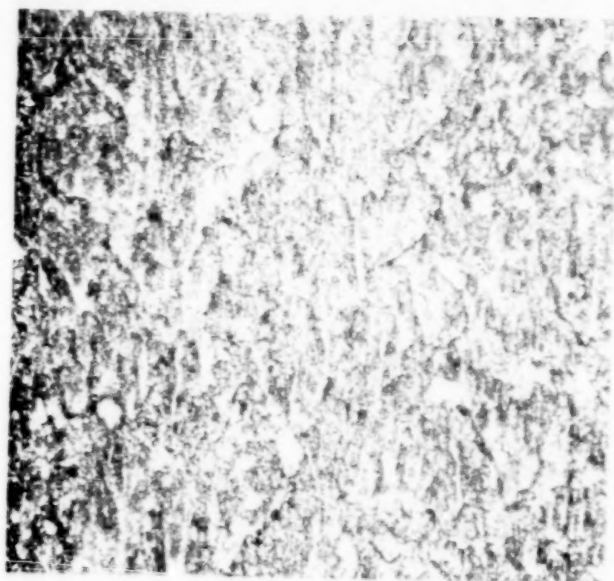


(b) Sanded substrate.

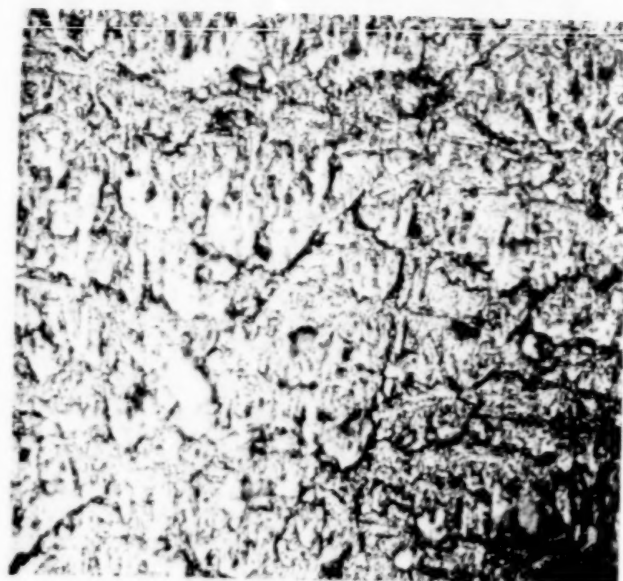


(c) Sandblasted substrate.

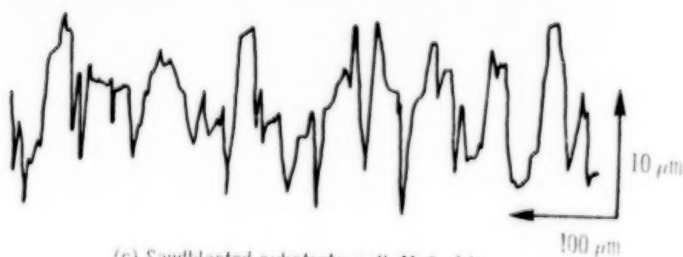
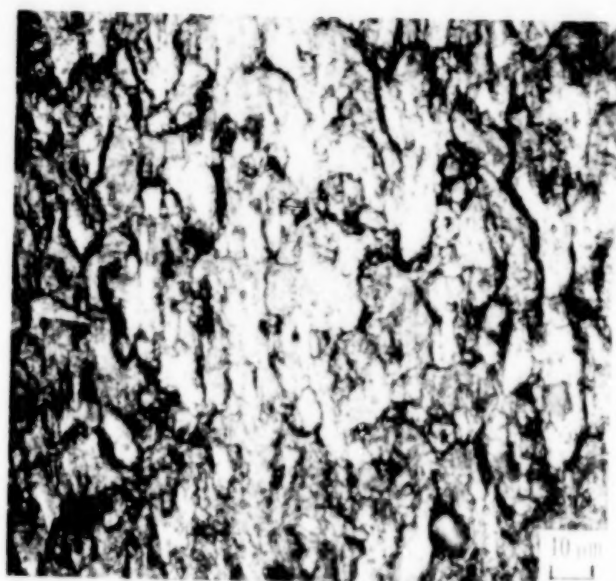
Figure 3. - Photomicrographs and surface profiles of 440C HT stainless-steel disks with different surface finishes before application of rubbed MoS_2 films.



(a) Polished substrate with MoS₂ film.



(b) Sanded substrate with MoS₂ film.



(c) Sandblasted substrate with MoS₂ film.

Figure 4. - Photomicrographs and surface profiles of 440C HT stainless-steel disks with different surface finishes after application of rubbed MoS₂ films.

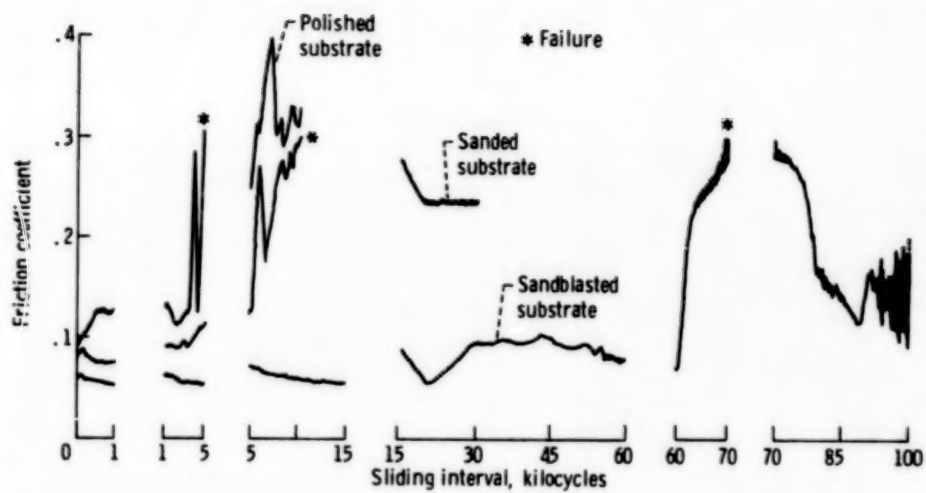
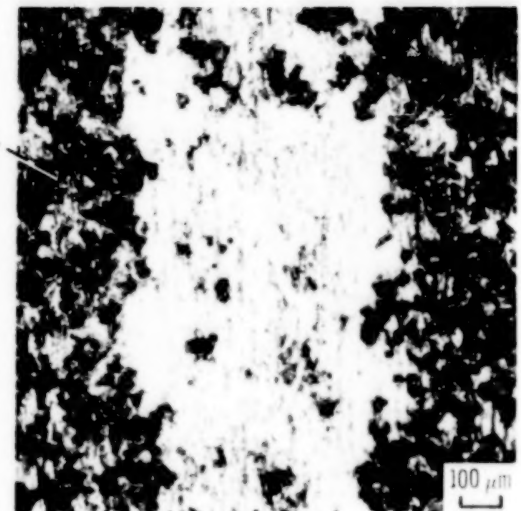
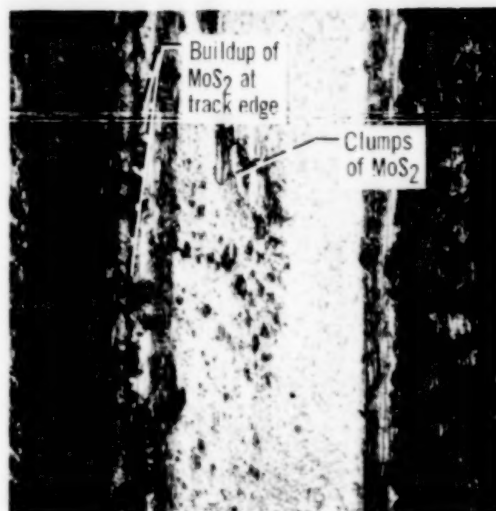
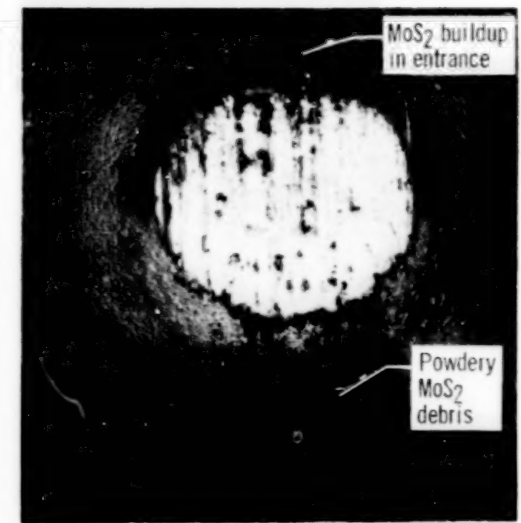
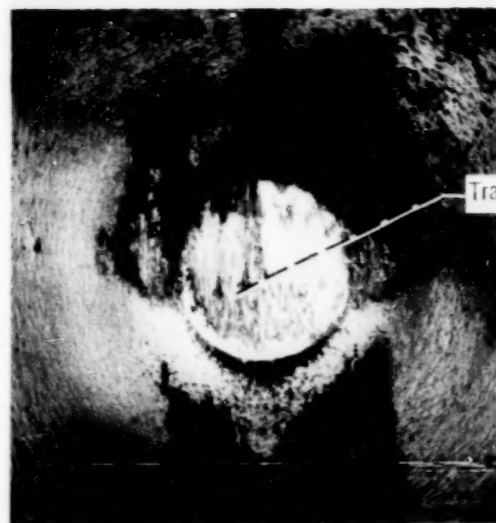


Figure 5. - Friction coefficient in moist air (10 000-ppm H_2O) as a function of kilocycles of sliding for rubbed MoS_2 films applied to 440C H1-stainless-steel disks with different surface finishes.

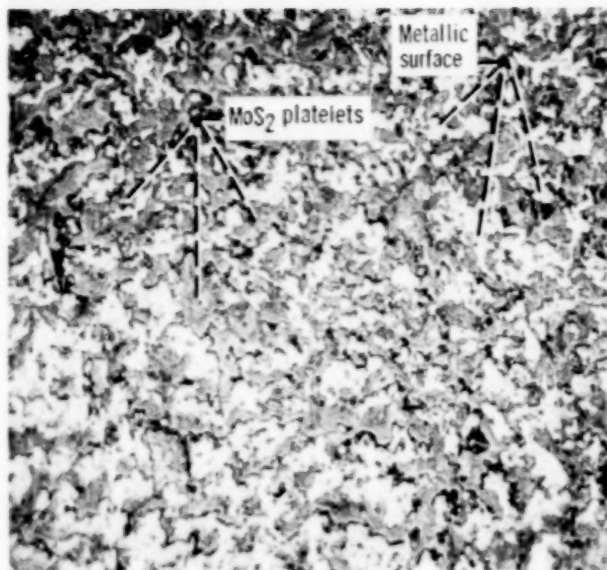


(a) Polished substrate.

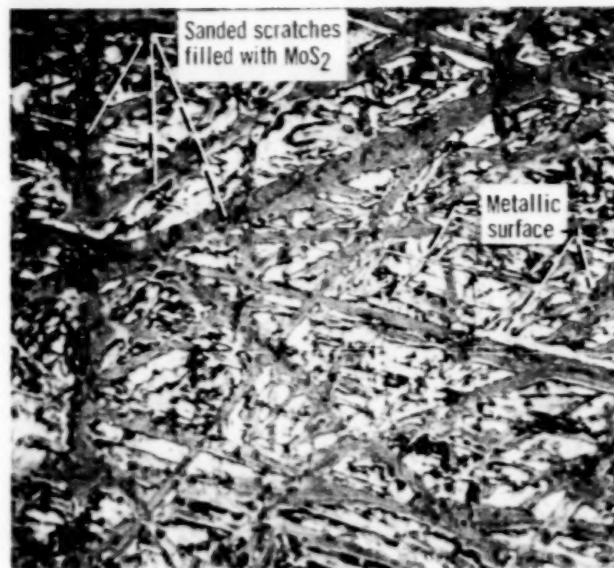
(b) Sandblasted substrate.

(c) Sandblasted substrate.

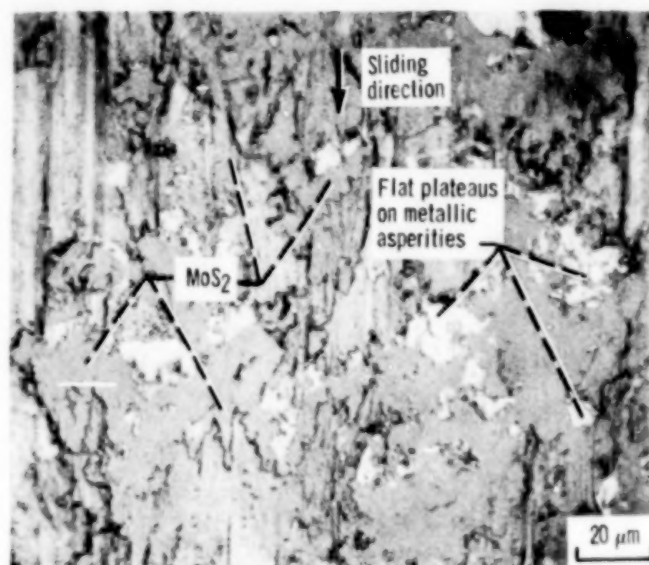
Figure 6. - Photomicrographs (taken after 1 kilocycle of sliding in moist air (10 000-ppm H_2O)) of rider wear scars and wear tracks on rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.



(a) Polished substrate.

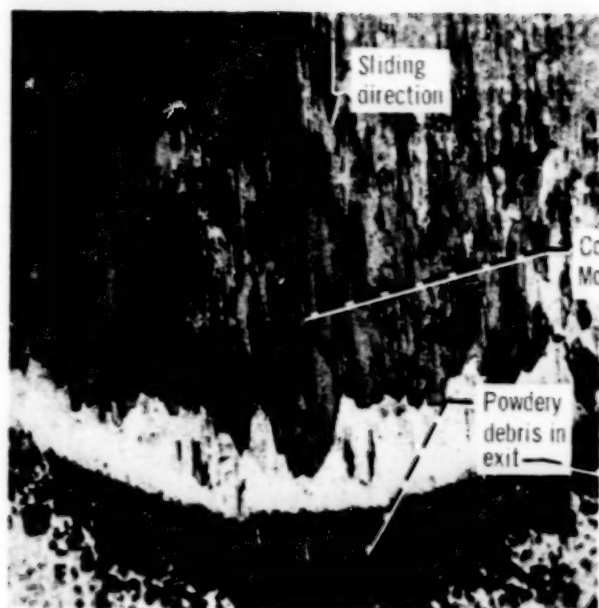


(b) Sanded substrate.



(c) Sandblasted substrate.

Figure 7. - High-magnification photomicrographs (taken after 1 kilocycle of sliding in moist air (10 000-ppm H_2O)) of wear tracks on rubbed MoS_2 films applied to 440C HT steel-stainless disks with different surface finishes.



(a) Polished substrate (exit region).

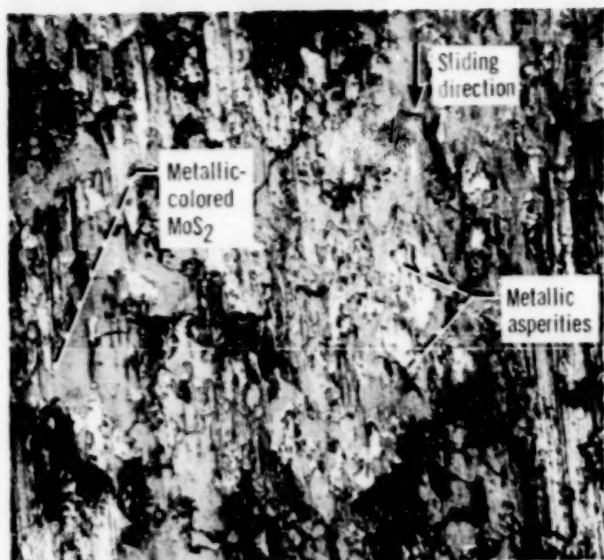


(b) Sanded substrate (exit region).

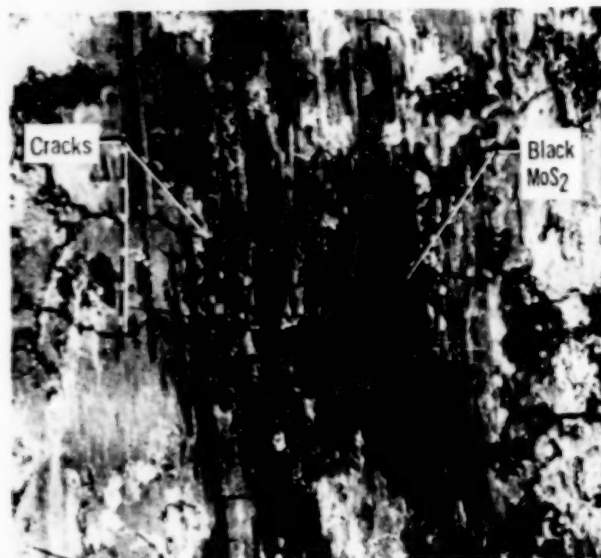


(c) Sandblasted substrate (entrance region).

Figure 8. - High-magnification photomicrographs (taken after 1 kilocycle of sliding in moist air (10 000-ppm H_2O)) of transfer films on riders that slid on rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.



(a) After 5 kilocycles of sliding.



(b) After 15 kilocycles of sliding.



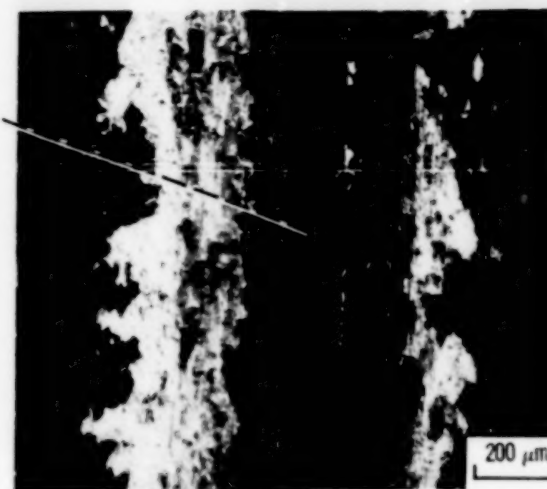
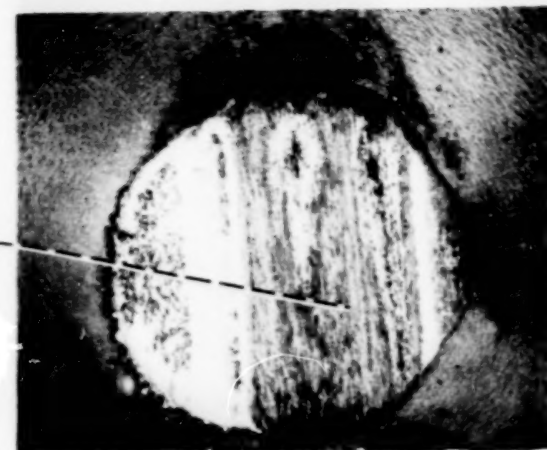
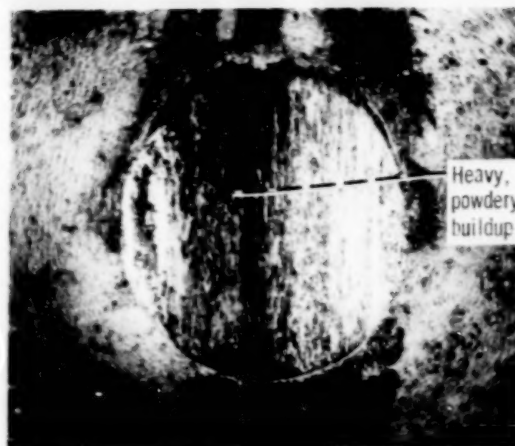
(c) After 60 kilocycles of sliding.



(d) After 70 kilocycles of sliding.

Figure 9. - High-magnification photomicrographs (taken after various intervals of sliding in moist air (10 000-ppm H_2O)) of wear tracks on rubbed MoS_2 films applied to sandblasted 440C HT stainless-steel disk surfaces.

22



(a) Polished substrate (after 5 kilocycles of sliding).

(b) Sanded substrate (after 10 kilocycles of sliding).

(c) Sandblasted substrate (after 70 kilocycles of sliding).

Figure 10. - Photomicrographs (taken after failure in moist air (10 000-ppm H_2O)) of rider wear scars and wear tracks on rubbed MoS_2 films applied to 440C HT stainless-steel substrates with different surface finishes.

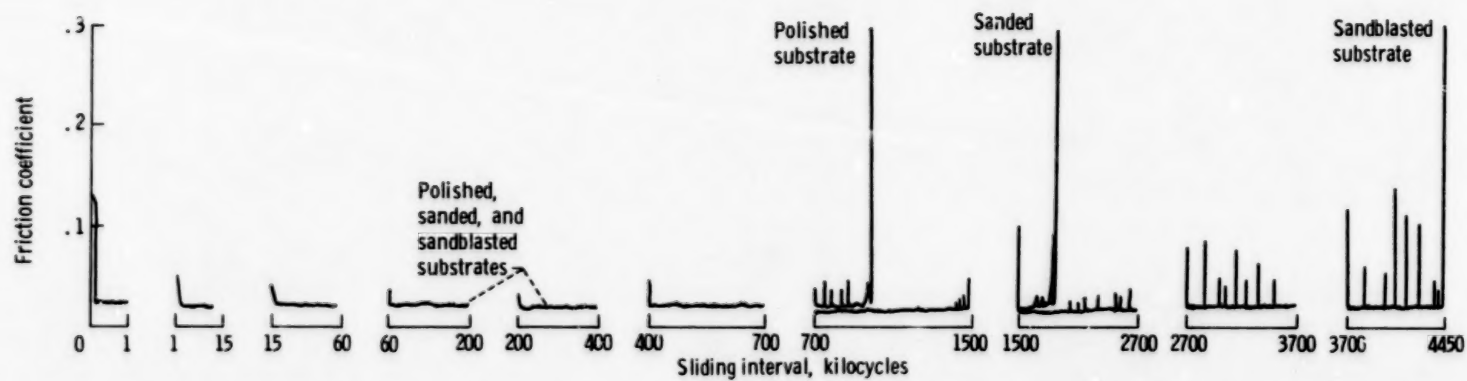
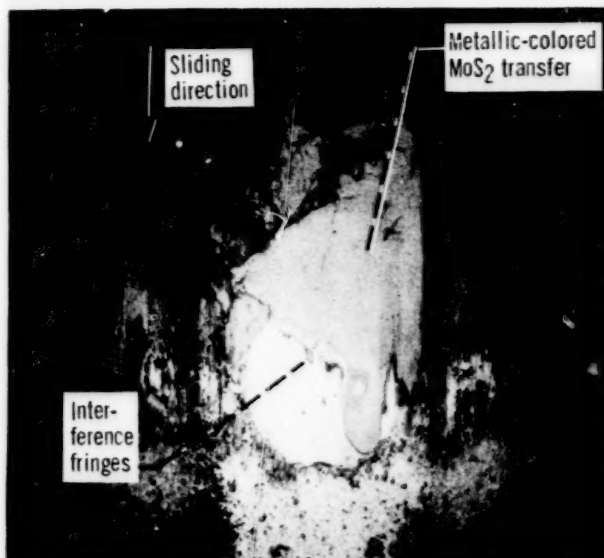
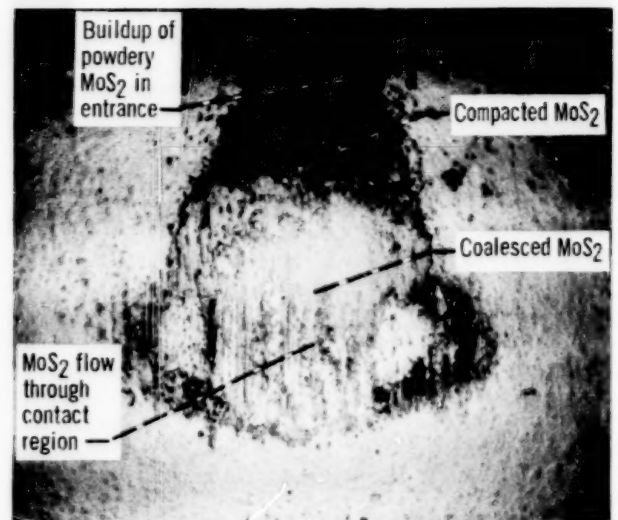


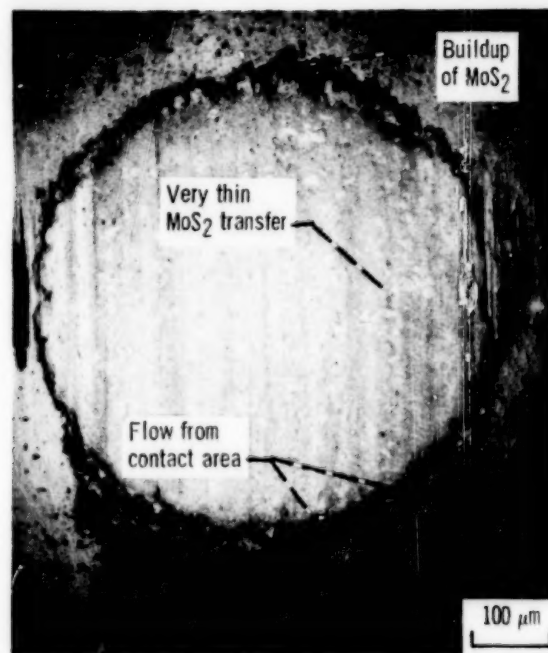
Figure 11. - Friction coefficient in dry argon (<20 -ppm H_2O) as a function of kilocycles of sliding for rubbed MoS_2 films applied to 440C HT stainless-steel disks with different surface finishes.



(a) Polished substrate.

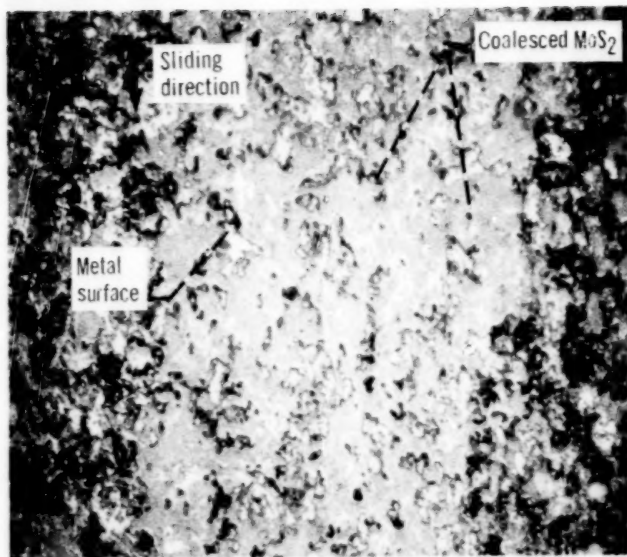


(b) Sanded substrate.

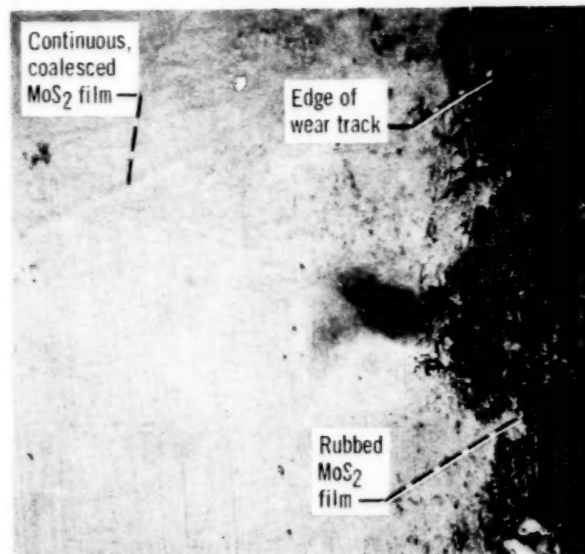


(c) Sandblasted substrate.

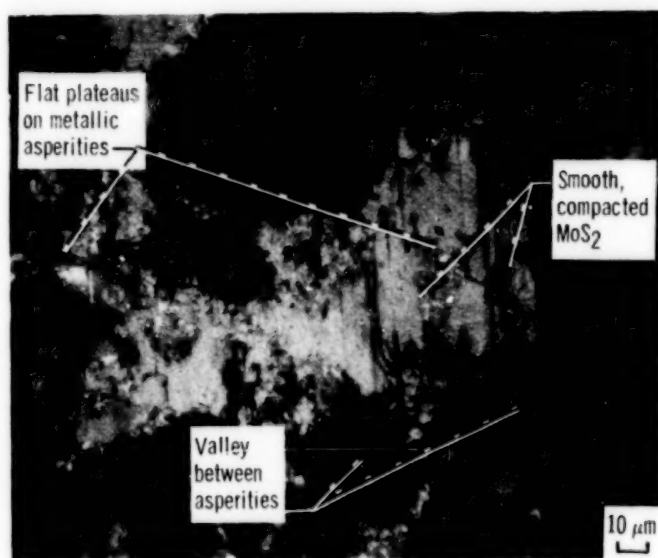
Figure 12. - Photomicrographs (taken after 1 kilocycle of sliding in dry argon (< 20-ppm H₂O)) of wear scars and transfer films on riders that slid on rubbed MoS₂ films applied to 440C HT stainless-steel disks with different surface finishes.



(a) Polished substrate.

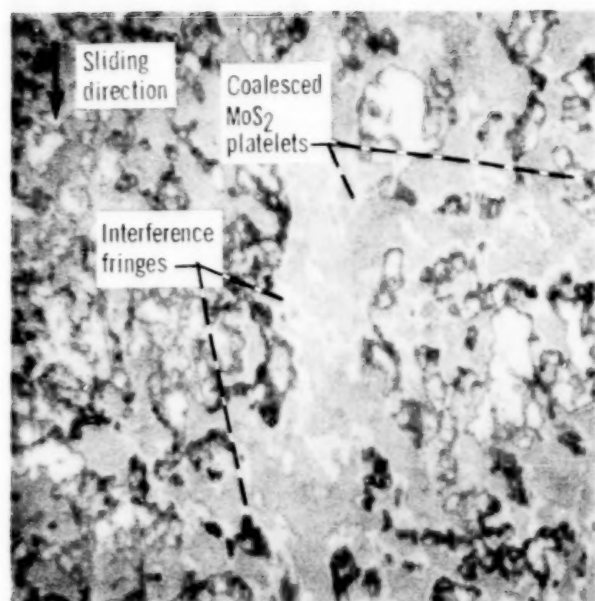


(b) Sanded substrate.

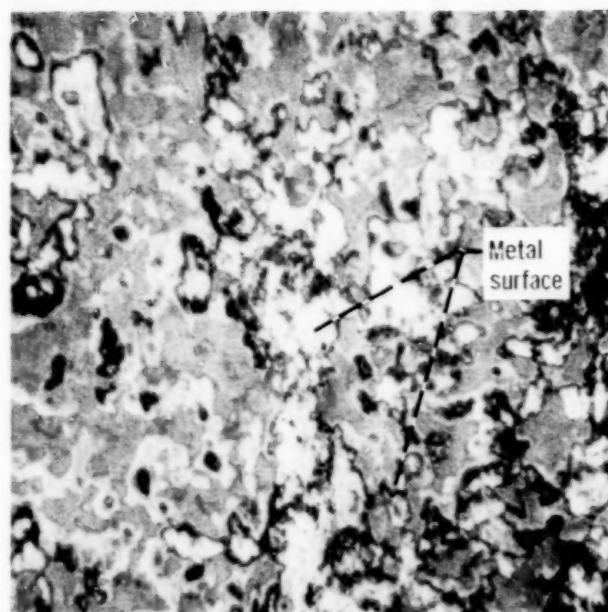


(c) Sandblasted substrate.

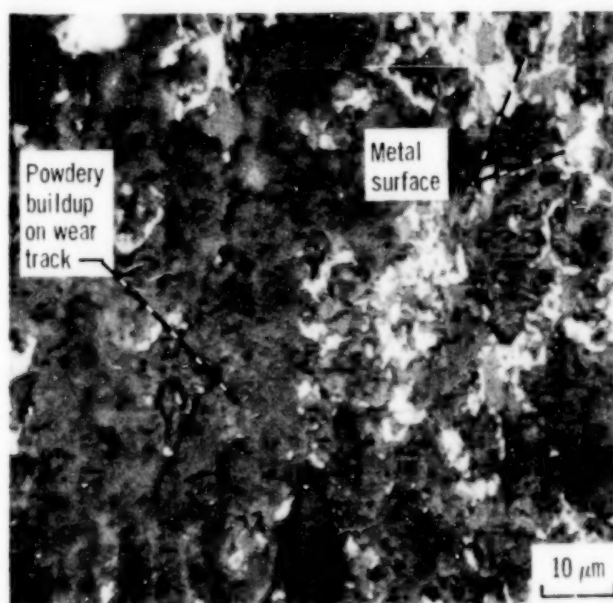
Figure 13. - High-magnification photomicrographs (taken after 1 kilocycle of sliding in dry argon (< 20-ppm H₂O)) of wear tracks on rubbed MoS₂ films applied to 440C HT stainless-steel disks with different surface finishes.



(a) After 60 kilocycles of sliding.



(b) After 700 kilocycles of sliding.



(c) After 940 kilocycles of sliding.

Figure 14. - High-magnification photomicrographs (taken in dry argon ($< 20\text{-ppm H}_2\text{O}$) after various intervals of sliding) of wear tracks on rubbed MoS_2 films applied to polished 440C HT stainless-steel disk surfaces.

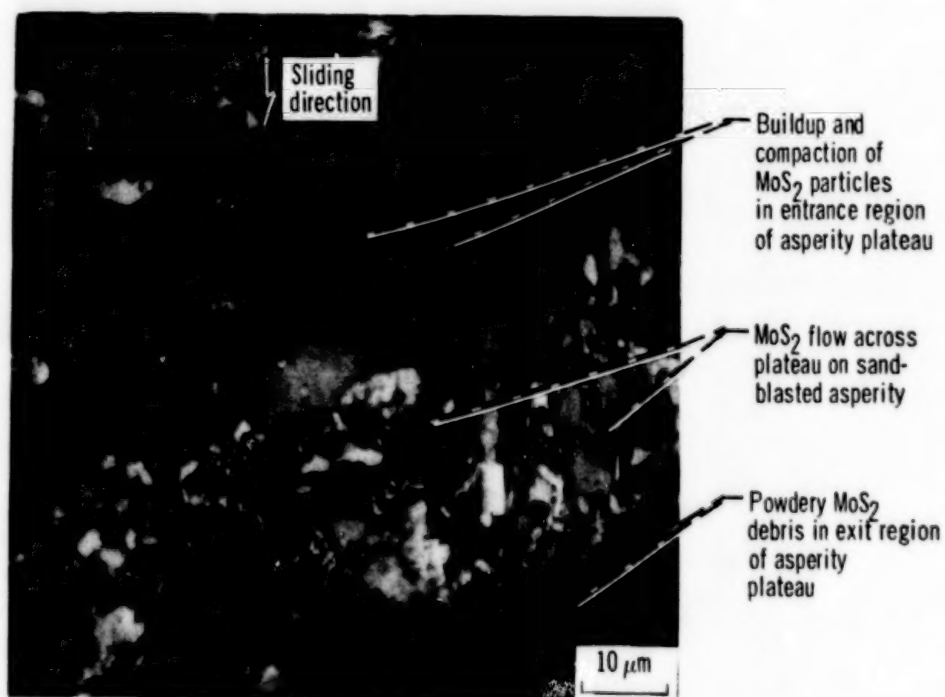


Figure 15. - High-magnification photomicrograph illustrating lubrication mechanism in dry argon (<20-ppm H₂O) of MoS₂ films applied to sandblasted 440C HT stainless-steel substrate surface.

1. Report No. NASA TP-1379		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle LUBRICATION AND FAILURE MECHANISMS OF MOLYBDENUM DISULFIDE FILMS II - EFFECT OF SUBSTRATE ROUGHNESS				5. Report Date December 1978	
				6. Performing Organization Code	
7. Author(s) Robert L. Fusaro				8. Performing Organization Report No. E-9715	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				10. Work Unit No. 505-04	
				11. Contract or Grant No.	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546				13. Type of Report and Period Covered Technical Paper	
				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract <p>An optical microscope was used to study the lubrication and failure mechanisms of rubbed MoS₂ films applied to three substrate surface finishes - polished, sanded, and sandblasted - as a function of sliding distance. The lubrication mechanism was the plastic flow of thin films of MoS₂ between flat plateaus on the rider and on the metallic substrate. If the substrate was rough, flat plateaus were created during "run-in" and the MoS₂ flowed across them. Wear life was extended by increasing surface roughness since valleys in the roughened substrate served as reservoirs for MoS₂ and as deposit sites for wear debris. In moist air the failure mechanism was the transformation of metallic-colored MoS₂ films to a black, powdery material that was found by X-ray diffraction to be α-iron, MoO₃, and possibly FeMoO₃. In dry argon the failure mechanism was the gradual depletion of MoS₂ from the contact region by transverse flow, and the wear debris on the track at failure was α-iron, residual MoS₂, and possibly FeS.</p>					
17. Key Words (Suggested by Author(s)) Molybdenum disulfide; Lubrication mechanisms; Friction and wear; Transfer films; Atmospheric effects on solid lubricants; Substrate roughness; Failure mechanisms of solid lubricants			18. Distribution Statement Unclassified - unlimited STAR Category 27		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 29	
				22. Price* A03	

90

50

END

MAR 16 1979